Fayalite content of olivine determined by reflectance measurements

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Procedure for determining the fayalite content by reflectance meansurements is given. Measurements in both vertical and oblique illumination are discussed. In vertical illumination an accuracy of ± 4 mol % fayalite can be obtained for Mg-rich olivines. The fayalite contents found are compared with values found by other methods, x-ray, microprobe or values found in literature.

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The accuracy of refractive index values for transparent minerals determined by means of reflectance measurements is generally questioned because of internal reflections in these minerals.

In 1971, however, Medenbach & Caye demonstrated that internal reflections can be avoided by using oblique illumination with an angle of incidence greater than 20°. Under these circumstances Fresnel's equations can be used to calculate the refractive index if the angle of incidence is known.

$$R_{\perp}^{i} = \left[\frac{\frac{\sqrt{1-\sin^2 i}}{n} - \cos i}{\frac{\sqrt{1-\sin^2 i}}{n} + \cos i} \right]^2 \quad (1)$$

Where $R_{\perp}^{i}$ is wave normal perpendicular to plane of incidence at an angle of incidence of i, and n is refractive index

Another way of calculating the refractive index is to take the average of two measurements of reflectance value carried out with the wave normal

1) perpendicular to the plane of incidence
2) parallel to the plane of incidence.

The average of these two measurements gives the reflectance value for normal incidence, as long as the angle of incidence does not exceed 25° (equation 2)

$$R^{i=o} = \frac{R_{\perp}^{i=22} + R_{\parallel}^{i=22}}{2} \quad (2)$$

where $R_{\perp}^{i=22}$ is the reflectance value at an angle of incidence of 22° and the wave normal is perpendicular to the plane of incidence.

The angle of incidence was controlled by an 'opal glass cube' with graduations incided on one side, which enabled an accuracy of the angle of incidence of about ± 2°. An accuracy of this order of magnitude is insufficient for the Fresnel calculation of the refractive index (eq. 1) for use in determining the fayalite content of olivine. Therefore the second method (eq. 2) has been used.

Materials studied

The reflectance value of olivine has been measured in the following 14 samples:

1 sample of fayalite from Rockport, Mass. USA

2 rocks from the Ilimaussaq intrusion, South Greenland. An augite syenite consisting of alkali feldspar, pyroxene (ferrosalite), fayalite and magnetite, and a heterogeneous foyaite composed of alkali feldspar, pyroxene (hedenbergite aegirine), fayalite, magnetite and nepheline (Lotte Melchior Larsen pers. comm.)

2 rocks from the Skærgård intrusion, East Greenland. One of them a ferrohortonolite ferrodiorite from upper zone b of the layered series and the other one a fayalite ferrodiorite from upper zone c of the layered series.

The remaining 9 samples are hornblende-peridotites from the Amitsoq intrusion, South Greenland.

Very small oxide inclusions, barely discernable with a × 16 objective, are present in most of the olivine grains, and it is necessary to carefully examine areas of measurement using a ×44 objective.

Two of the samples, the fayalite ferrodiorite and the hornblende-peridotite contained so many oxide inclusions that it was difficult to find areas large enough for measurement.

Measurements

The reflectance value of olivine was measured in vertical as well as in oblique illumination with optical conditions as in table 1. The measurements were carried out at 590 NM and SICA was used as standard.

Table 1

	vertical ill.	oblique ill.
objective	16/0.40	44/0.85
area of stage object measured	20 × 20 μm	7 × 7 μm
aperture of ill.	approx. 3°	approx. 7°

As the measurements progressed it became clear, that one of the most critical conditions in measuring in oblique illumination was obtaining exact focus.

For vertical incidence Piller & v. Gehlen (1964) have shown that insufficient mechanical stability gives rise to increasing errors with rising magnification.

Using ×44 objective, the influence of defocussing 1μm and the relative change in reflectance values of olivine were determined for varying angles of incidence (table 2).

Table 2

angles of incidence i°	relative change in reflec. value of olivine by defocussing 1 μm – % –	changes in fayalite content corresponding to defocussing 1 μm mol %
18	0.7	1.7
21.5	3.1	7.4
25	7.2	17.2
28.5	10.1	23.9

Table 2 shows that at an angle of incidence of 21.5° i.e. close to the lowermost angle of incidence which can be used if internal reflections are to be avoided, an inaccuracy in focussing of 1 μm, may cause an error in the determination of fayalite content of 7.4 mol %. Rotation of the microscope stage might easily cause defocussing of this magnitude.

This sensitivity with respect to defocussing is further illustrated in table 3 where the fayalite content found by reflectance measurements in vertical and oblique illumination are compared. The values presented in table 3 were obtained without refocussing after rotation of the microscope stage. The reason for rotating the stage is that the highest and lowest reflectance value of the sections have been used to calculate the mean reflectance value. Further details concerning this will be given below.

Table 3 also shows the fayalite content found by x-ray diffraction and the deviation of the fayalite contents between those found by x-ray diffraction and those found by reflectance values for vertical and oblique illumination.

The values in table 3 show that if measurements are made without refocussing, vertical illumination using ×16 objective is more accurate than oblique illumination using ×44 objective.

The values also show that one must expect at least 1μm in defocussing when rotating the microscope stage, due to the mechanical properties in the combined system of microscope and specimen.

It is therefore necessary to refocus over and over again to find the true values of the sections in question. This is, however, a too laborious method for routine work, and in many

Table 3

No.	Fayalite contents in mol % found by Refl. i=0°	Refl. i=22°.5	x-ray	Deviations A	AA
133366	18.2	14.2	19.0	-0.8	- 4.8
133367	14.7	24.4	16.1	-1.4	8.3
133368	19.7	24.2	17.1	2.6	7.1
133369	22.7	15.1	28.2	-5.5	-13.1
133370	19.0	25.0	17.2	1.8	7.8
133371	21.9	16.1	25.7	-3.8	- 9.6

A. deviations of the fayalite contents found by refl. measurements with i=o from those found by x-ray diff.
AA. deviations of the fayalite contents found by refl. measurements with i=22°.5 and those found by x-ray diff.

cases conventional immersion methods or x-ray diffraction are preferable.

Determinations of the fayalite content with a margin of error of about ± 4 mol %, or at a maximum ± 5 mol % can, however, be made in vertical illumination by carefully selecting areas without internal reflections. In some cases an accuracy of that order may be sufficient for characterizing rocks.

In the following, the procedure used in determining the fayalite content by reflectance measurements in vertical illumination will be given.

In each sample 10 grains were measured. The highest (R_1) and lowest (R_2) reflectance values for each grain were found, and the average reflectance value ($\bar{R}$) for each sample was calculated as follows:

$$\frac{R_1 + R_2}{2} = R_a, \quad \frac{R_a + R_b + \cdots R_j}{10} = \bar{R} \quad (3)$$

The determination of the fayalite content is based on the assumption that randomly oriented olivine grains have an average refractive index corresponding to

$$\bar{n} = \tfrac{1}{3}(n_\alpha + n_\beta + n_\gamma) \quad (4)$$

which is equivalent to an average reflectance value of

$$\bar{R} = \tfrac{1}{3}(R_\alpha + R_\beta + R_\gamma) \quad (5)$$

This seems reasonable because the optical angles for most of the olivines are close to 90°.

The principal reflectance value of olivine was found by inserting the refractive index values given by Deer, Howie & Zussmann (1962) into equation (6).

$$R_v = \frac{(n_v - 1)^2}{(n_v + 1)^2}, \quad v = \alpha, \beta, \gamma. \quad (6)$$

By inserting the principal reflectance values in equation (5) the following linear correspondance between average reflectance value and mol % fayalite was established

$$\bar{R} = 6.04 + \tfrac{1}{100}(9.01 - 6.04)\,(\text{mol } \%\ \text{Fa}) \quad (7)$$

This equation is outlined in fig. 1.

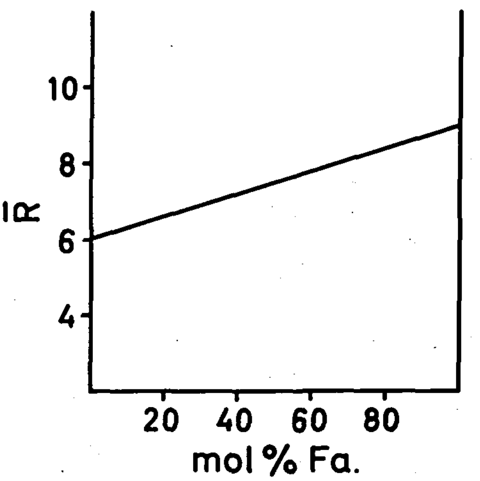

Fig. 1.

In table 4, the fayalite contents found by reflectance measurements are compared with the fayalite contents found by x-ray diffraction or microprobe analyses. Also some values taken from the literature are shown.

Determinations by x-ray diffraction were carried out using a Guinier-Hägg camera with LiF as an internal standard, and the Yoder & Sahama (1957) equation has been used to calculate the fayalite content.

The microprobe analyses were carried out by Lotte Melchior Larsen at the Geological Central Institute at the University of Copenhagen. The microprobe values in table 4 are average values of analyses taken from core and

Table 4

	Samples of rock		*Composition of olivine (mol % fayalite)* measured by:			found in	
No.	Type	Location	Refl. values	Micro-probe	X-ray diffr.	Literature	Deviations
49/74	Fayalite	Rockport, Mass. USA	103.0			98.3 x)	
50/74	Fayalite	Rockport, Mass. USA	102.4			Fa + Tph	
91943	Fayalite in foyaite	Ilimaussaq intrusion Greenland	101.9	98.6			3.3
91939	Fayalite in augite-syenite	Ilimaussaq intrusion Greenland	92.6	89.8 Fa + Tph			2.8
53/74	Fayalite ferrodiorite Layered series Upper zone c	Skærgård intrusion Greenland	96.6			98 – 87 xx)	
52/74	Ferrohortonolite ferrodiorite Layered series Upper zone b	Skærgård intrusion Greenland	73.1			71.5 – 87 xx)	
133366	Chrysolite in horn-blende-peridotite	Amitsoq intrusion Greenland	18.2		19.0		– 0.8
133367	»	»	14.7		16.1		– 1.4
133368	»	»	19.7		17.1		2.6
133369	»	»	22.7		28.2		– 5.5
133370	»	»	19.0		17.2		1.8
133371	»	»	21.9		25.7		– 3.8
133373	»	»	15.9		19.6		– 3.7
133374	»	»	20.8		20.3		0.5
133375	»	»	22.7		26.0		– 3.3

x) Fa + Tph = Fayalite + Tephorite content, values taken from Bowen & Posnjak (1933)
xx) Values taken from Wager & Brown (1967)

border of the olivine grains. Further the values are the sum of fayalite and tephorite contents.

The determination of the fayalite content using Yoder & Sahama's equation is subject to an error of 3 mol % for Mg-rich olivines. This error is of the same order of magnitude as the difference between the fayalite content found by reflectance measurement and that determined by x-ray diffraction (see table 4). On the basis of this one can estimate the accuracy of the fayalite content by reflectance measurement to be about ± 4 mol % for Mg-rich olivines.

Owing to their optical angle, the average reflectance value of Fe-rich olivines deviates more than those of Mg-rich when equation (5) is used. For Fe-rich olivines errors of ± 5 mol % or more must be expected.

However, a much more important error concerning natural Fe-rich olivines is due to their normally high content of manganese. The determinations of fayalite content by reflectance measurement are based on the assumption that only Fe and Mg occur in the olivines.

Therefore when working with natural olivines the accuracy in determining the fayalite content of Fe-rich olivines depends more on the Mn-content than on the measuring procedures.

Conclusions

If areas free from internal reflections and oxide inclusions are carefully selected it is possible to determine the fayalite content of Mg-rich olivines with an accuracy of ± 4 mol % by means of reflectance measurements in vertical illumination.

The same method of determination applied to Fe-rich olivines leads to an estimated accuracy of ± 5 mol %. This is, however, only true for Fe-rich olivines without Mn.

In the case of anisotropic minerals like olivine it must be questioned whether oblique illumination would provide better accuracy in determining the refractive index than vertical illumination. The fact is that the accuracy of the determination of the average refractive index of anisotropic minerals depends on the accuracy with which the average reflectance value is determined. This again is a question of the number of grains measured rather than conjecture of the influence of minor internal reflections in some few grains.

The discussed method of determining the refractive index can likewise especially be applied to minerals with high refractive indices such as most heavy minerals, or to samples where the mineral occurs either as small grains or in small quantities.

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Dansk sammendrag

Ved hjælp af reflektansmålinger i vertikal belysning er det muligt at bestemme Mg-rige oliviners fayalit-indhold med en nøjagtighed på ±4 mol % fayalit. Nøjagtigheden for Fe-rige oliviner ligger omkring ±5 mol %, forudsat at Mn-indholdet er beskedent.

Det ved hjælp af reflektansmålinger fundne fayalit-indhold sammenlignes i tabel 4 med fayalit-indholdet fundet enten ved hjælp af røntgen-optagelser, mikrosonde-analyser eller værdier taget fra litteraturen.

Reflektansmålinger er særlig velegnet til at bestemme lysbrydningen af mineraler med høj lysbrydning som for eksempel mange tungmineralers lysbrydning.

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